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**TECHNICAL REPORT ARLCD-TR-77010** 

# A TECHNIQUE FOR ASSESSING THE DURABILITY OF STRUCTURAL ADHESIVES

RAYMOND F. WEGMAN MARIE C. ROSS ELIZABETH A. GARNIS STANLEY A. SLOTA



**MAY 1978** 



US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
LARGE CALIBER
WEAPON SYSTEMS LABORATORY
DOVER, NEW JERSEY

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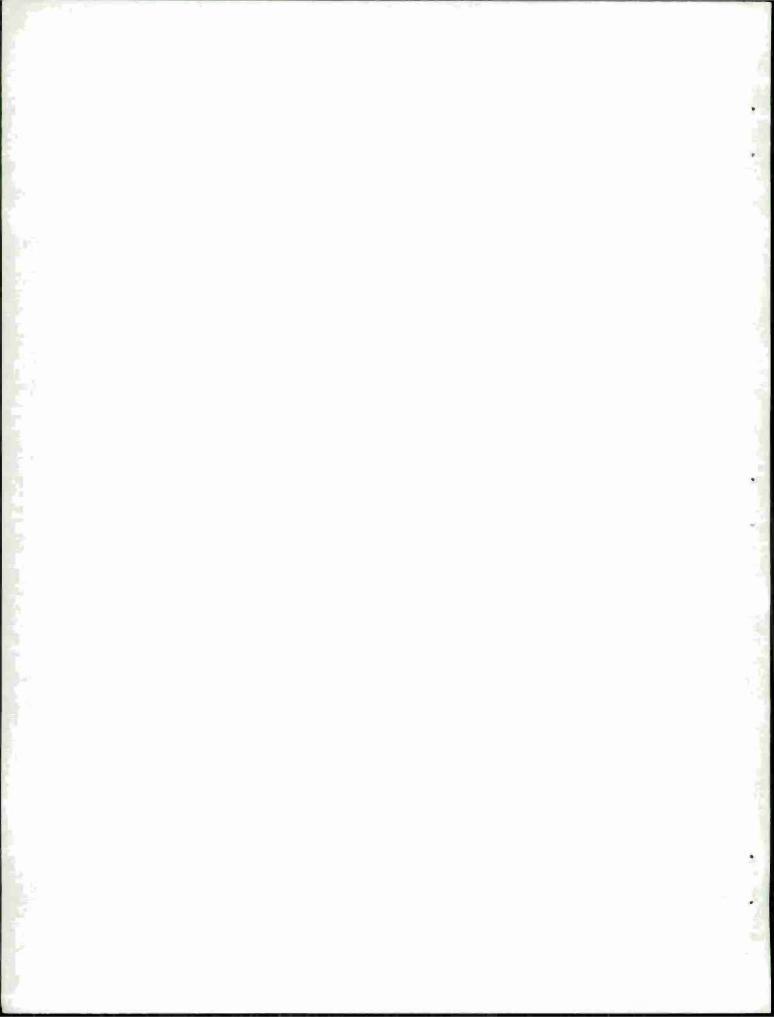
# 20. ABSTRACT (Continued)

to select the best adhesives and adherend surface treatments for a particular application. The method involves determining the residual strength after the bonded joints are immersed in 60°C water for prescribed periods of time.

Data are presented for twelve structural adhesives which are 121°C (250°F) curing systems. The adherends used were 2024T3 aluminum, either acid-dichromate (FPL) etched or anodized, 6 A1-4V titanium and commercially pure (CP) titanium, both phosphate-fluoride etched.

# **ACKNOWLEDGMENT**

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24 Effect of -20°C storage of the adhesive on the durability of bonds tested by using the 1000 hour, 60°C water-soak residual-strength values

#### INTRODUCTION

The durability of adhesive-bonded joints has been evaluated a number of times (refs. 1-30). One method used (ref. 26) involves applying stress to the joint and subsequently exposing the stressed joint to a test environment. This is expensive and time consuming because it requires environmental test chambers.

A new test has been developed (ref. 31) in which a large number of specimens can be conditioned unstressed, simultaneously, and tested for residual strength. The results can be compared to rank the adhesive joints in order of durability. By combining this new test with the data for a single stress level test, a prediction curve for the behavior of the joints under the ASTM D-2919-71 test can be prepared (ref. 31-34).

#### DISCUSSION

The twelve adhesive systems used in this investigation are listed in table 1. All are 121°C (250°F) curing systems. The adherends and their surface preparations are as follows:

- 2024-T3 aluminum alloy, acid-dichromate (FPL) etched or anodized.
- 2. Commercially pure (CP) titanium alloy, phosphate-fluoride etched.
  - 3. 6 A1-4V titanium alloy, phosphate-fluoride etched.

References 32 and 33 show that the hot-water soak test gives a pattern of results similar to those obtained from the stressed durability tests for 121°C (250°F) adhesives. Plotting the time-to-failure data obtained at one stress level at 60°C and 95% relative humidity (RH) and the hot-water soak/residual strength data on the same graph, and drawing a line parallel to the hot-water soak line, yields a prediction curve for the stressed durability test (fig. 21). The prediction curve origin is taken at the average of the time-to-failure at the stress level chosen.

Figures 1 through 23 summarize graphically all the data obtained in this program. Most of the graphs contain hot-water soak and stressed durability curves along with a prediction curve. Figures 21 through 23 contain only the hot-water soak curve and a prediction curve based on one set of time-to-failure data. The slope of the actual curve may vary from the slope of the predicted curve; most of the differences are considered within experimental error due to the scatter expected in adhesive mechanical data. Reference 35 shows that a reasonable estimate of lifetime can be obtained from hot-water aging data for some of the adhesive systems which were studied in this investigation.

Some comments on the individual plots are given in table 2.

For each set of data, the stressed-durability test is generally quicker than the hot-water soak test. If a large number of adhesive-adherend combinations are to be tested, however, the hot-water soak test will be faster and will cost less. Water-soak specimens can be conditioned without using stress fixtures, large environmental chambers, or extensive instrumentation. Only the temperature-controlled water bath and standard tensile testing equipment are required.

In reference 34, the hot-water soak/residual-strength test was used to monitor the storage lives of D, I, K, and L adhesives. The adhesives were stored at -20°C (-4°F) immediately after their manufacture and removed monthly for specimen preparation. Figure 24 is the graphic presentation of the results for the systems. The 1000 hour hot-water soak/residual-strength curve shows a substantial decrease for adhesive sample I at 9 months, indicating that the maximum useful storage life of the adhesive at -20°C is 8 months. For adhesives K and L, the storage life at -20°C was at least 13 months. Adhesive D slowly decreased in durability after 7 months storage.

#### EXPERIMENTAL PROCEDURES

#### Preparation of Adherends

The 2024-T3 aluminum alloy was etched with acid-dichromate (FPL) etch as described in reference 32. The anodized 2024-T3 aluminum was prepared as described in reference 32. The CP and 6 A1-4V titanium were prepared as described in reference 21.

# Methods of Testing

# Lap-Shear Tensile Strength

A Baldwin Universal Test Machine was used for load application. The load rate was 16.5 MPa (2400 psi)/minute.

# Hot-Water Soak/Residual-Strength

Adhesive-bonded, lap-shear specimens were immersed for a prescribed time in a thermostatically controlled tank at 60°C (140°F). The specimens were removed and placed in a 60°C (140°F) container of water. The water container, with the specimens inside, was placed in the test chamber of a Baldwin Tensile Test Machine at 60°C (140°F). The individual specimen to be tested was removed from the water container and placed in the test grips. A thermocouple was attached with adhesive tape. When the digital thermometer attached to the thermocouple registered 60°C, the specimen was tested to failure to determine its residual strength after the hot-water immersion.

# Stressed Durability

The stressed-durability testing was done in accordance with the basic method described in reference 26 except that the fixtures were equipped with a timing device to record the elapsed time the specimens were under test before failure. This timing device is descrived in reference 18.

# Mathematical Calculation of the Best Fit of Data for Durability Curves

The data was processed with a Hewlett Packard 9100A calculator using the Hewlett Packard program 09101-70803. This program calculates the equation of the straight line of best fit of a set of data points. The best fit is determined by minimizing the sum of the squares of the deviations of the data points from the line.

The program claculates m and b for the equation

$$Y = mX + b. (1)$$

The program also calculates a correlation coefficient r, an indication of goodness of fit. Note  $-1 \le r \le 1$  where the sign corresponds to the slope m. If r=0 there is no correlation, and if  $r=\pm 1$  there is perfect correlation or a perfect fit.

The defining equations taken from reference 36 are

$$m = \frac{\sum_{i=1}^{n} (X_{i} - \overline{X}) (Y_{i} - \overline{Y})}{\sum_{i=1}^{n} (X_{i} - \overline{X})^{2}}$$
(2)

$$b = \overline{Y} - m\overline{X} \tag{3}$$

where

$$\overline{Y} = \frac{\sum_{i=1}^{\infty} Y_i}{n} \tag{4}$$

and

$$\bar{X} = \frac{\sum_{i=1}^{n} X_i}{n} \tag{5}$$

$$r = \frac{\sum_{i=1}^{n} (X_{i} - \bar{X}) (Y_{i} - \bar{Y})}{\sqrt{\sum_{i=1}^{n} (X_{i} - \bar{X})^{2} \Sigma (Y_{i} - \bar{Y})^{2}}}$$
(6)

The program was adapted to change the x axis data to log x for convenient use with semilog graph paper.

#### Conversion to SI Units

Conventional stress units of pounds per square inch (psi) were converted to SI units by the following:

$$\frac{\text{psi} \times 6.8948}{1000} = \text{MPa}$$

This was in accordance with ASTM E 380-74, table 4.

#### CONCLUSION AND RECOMMENDATION

The hot-water soak/residual-strength test described in this report can inexpensively predict the stressed durability of many 121°C curing/adhesive-adherend combinations. This method needs only one set of stressed data for each combination.

Further evaluation should be made for other adhesive systems, especially those cured at temperatures above 121°C.

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Table 1. Adhesives

Code	Type
A	Supported, modified epoxy film
В	Supported, modified epoxy film
С	Supported, modified epoxy film
D	Supported, modified epoxy film
E	Unsupported, modified epoxy film
F	Supported, modified epoxy film
G	Supported, modified epoxy film
Н	Supported, modified epoxy film
t.	Supported, modified epoxy film
J	Supported, modified epoxy film
К	Supported, modified epoxy film
L.	Supported, modified epoxy film

Table 2. Comments on data plots

Figure Number	Comments
1	The prediction curve is almost identical to the stressed durability curve, with a slightly different slope.
2	The slope of the prediction curve is slightly different than the slope of the stressed durability curve.
3	The predicted time-to-failure is slightly lower than the actual time-to-failure.
4	The curves are almost identical.
5	Same as 3.
6	The prediction curve indicates longer time-to-failure at high loads and shorter time-to-failure at low loads than the stressed durability curve.
7	Same as 3.
8	Same as 3.
9	The prediction curve indicates shorter time-to-failure at high loads and longer time-to-failure at low loads than the stressed durability curve.
10	Same as 6.
11	Same as 2.
12	The prediction and stressed durability curves are identical.
13	Same as 3.
14	Same as 4.
15	Same as 2.

Table 2 (Continued)

Figure Number	Comments
16	Same as 4.
17	Same as 2.
18	Same as 2.
19	Same as 9.
20	Same as 9.
21,22,23	Stressed durability curve not determined; only a prediction curve based on one set of time-to-failure data is shown.

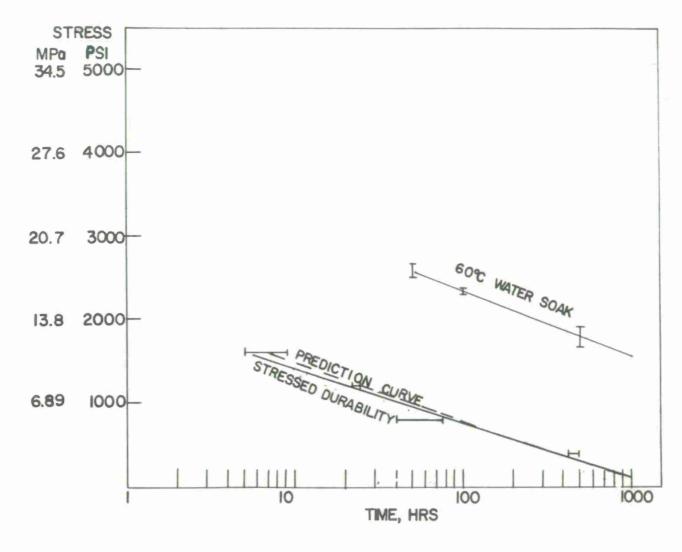


Figure 1. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive A

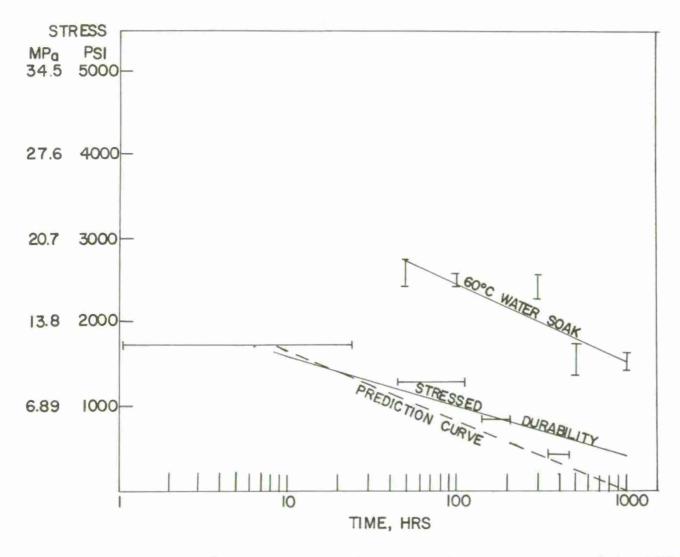


Figure 2. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for anodized 2024T3 aluminum alloy joints bonded with adhesive A

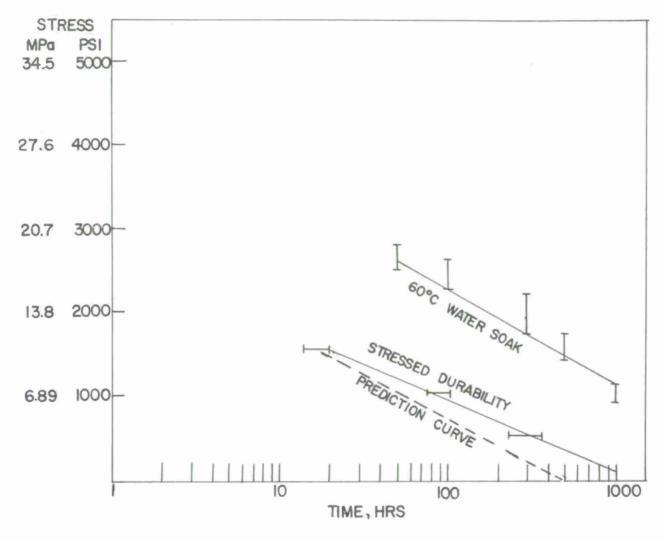


Figure 3. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive A

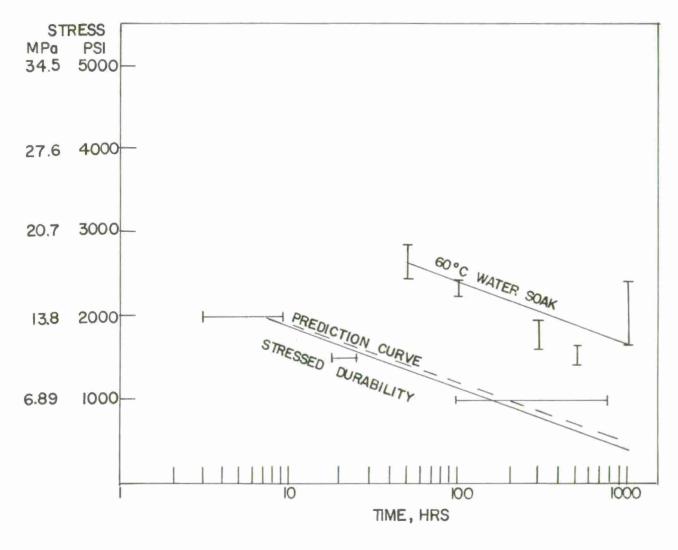


Figure 4. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched 6,4 titanium alloy joints bonded with adhesive A



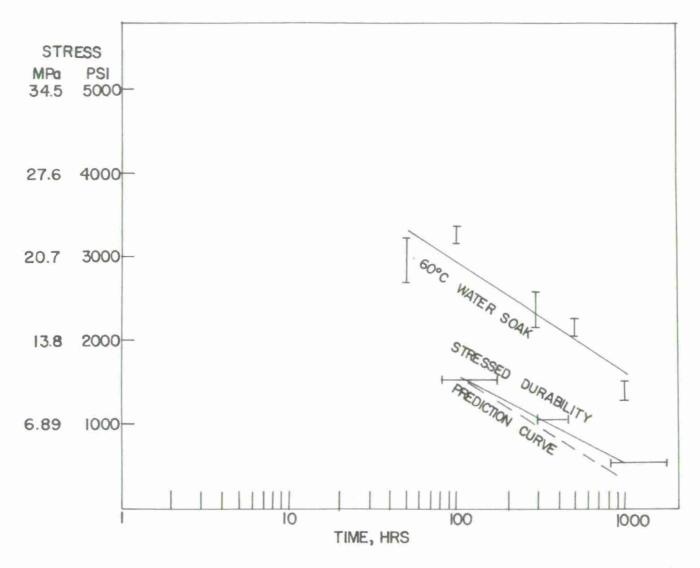


Figure 5. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive B

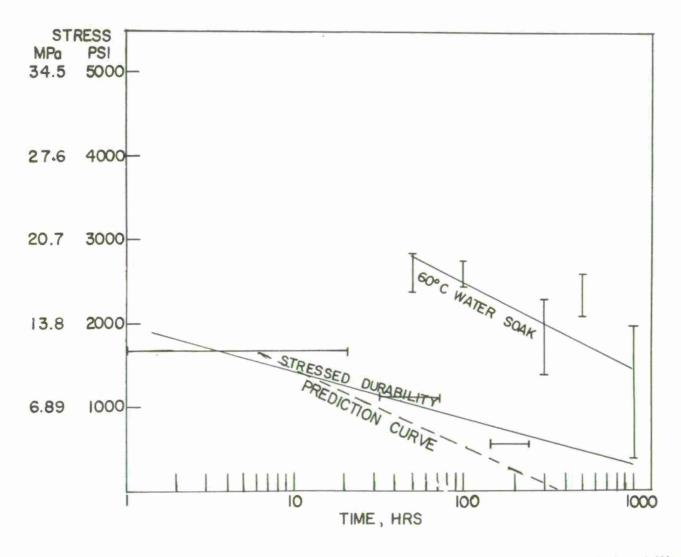


Figure 6. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched 6,4 titanium alloy joints bonded with adhesive C

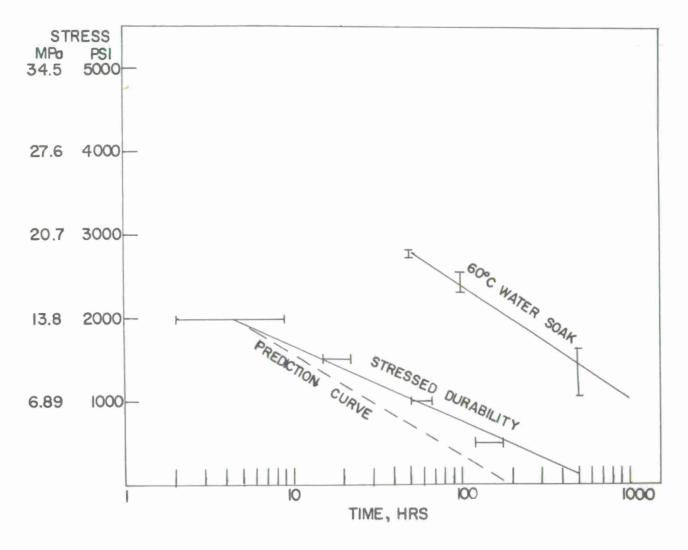


Figure 7. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive C

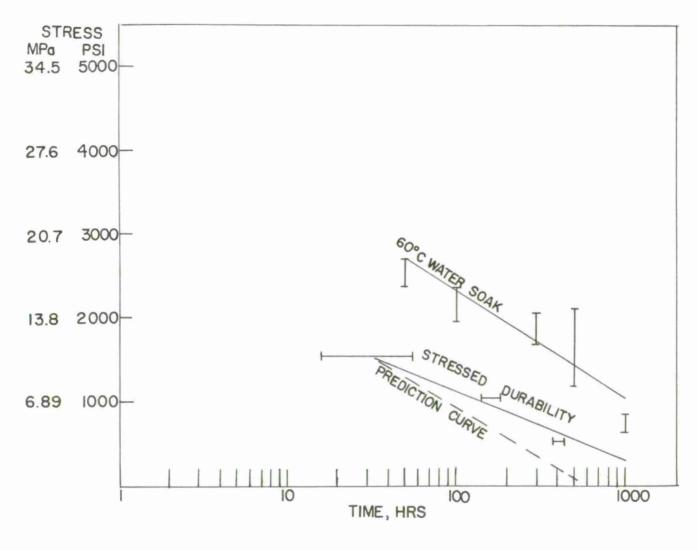


Figure 8. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive C

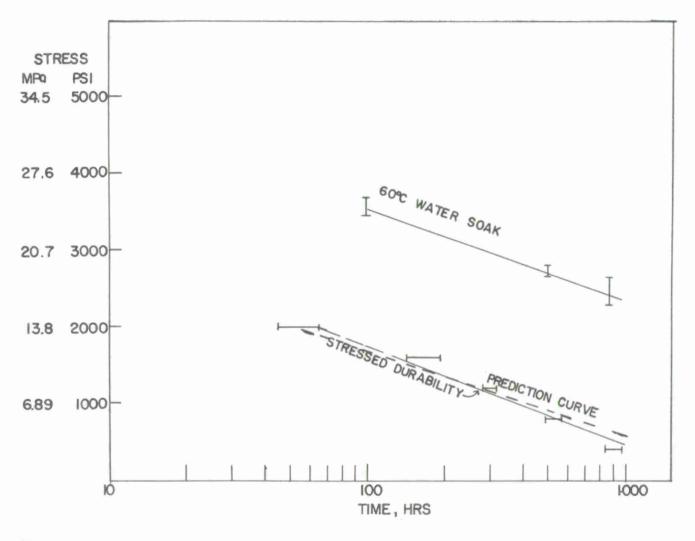


Figure 9. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive D

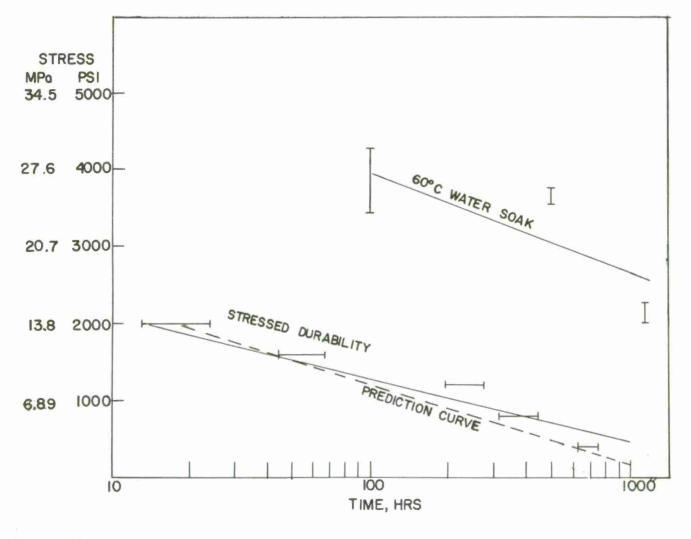


Figure 10. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for anodized 2024T3 aluminum alloy joints bonded with adhesive D

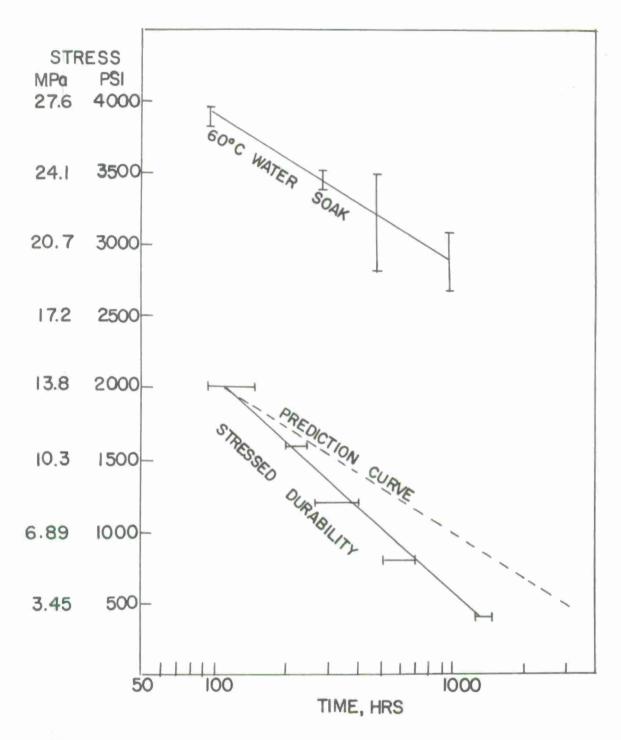


Figure 11. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive D

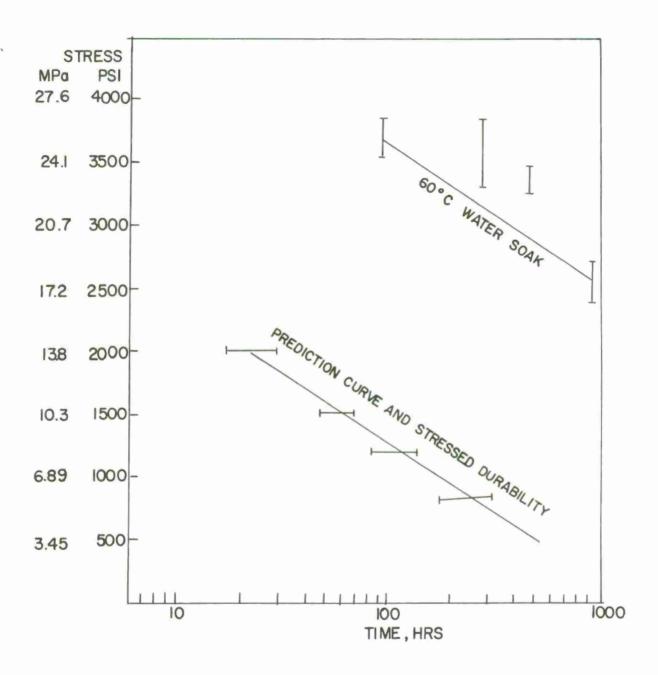


Figure 12. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive G

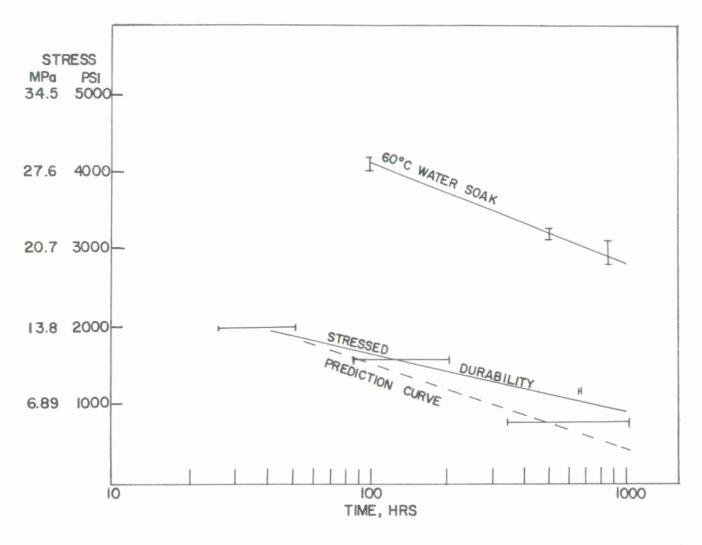


Figure 13. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive I

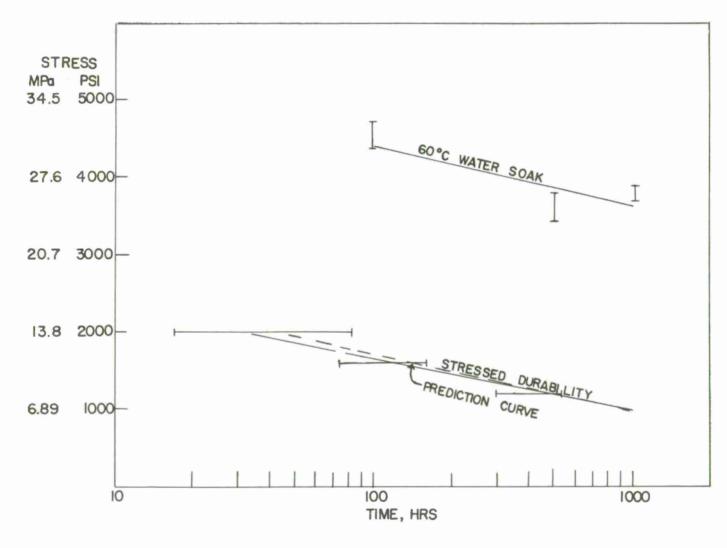


Figure 14. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for anodized 2024T3 aluminum alloy joints bonded with adhesive I

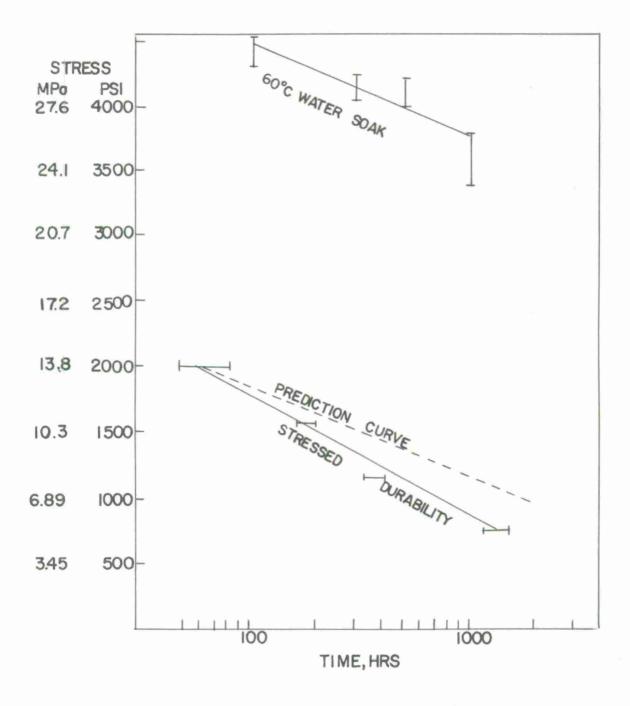


Figure 15. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive I

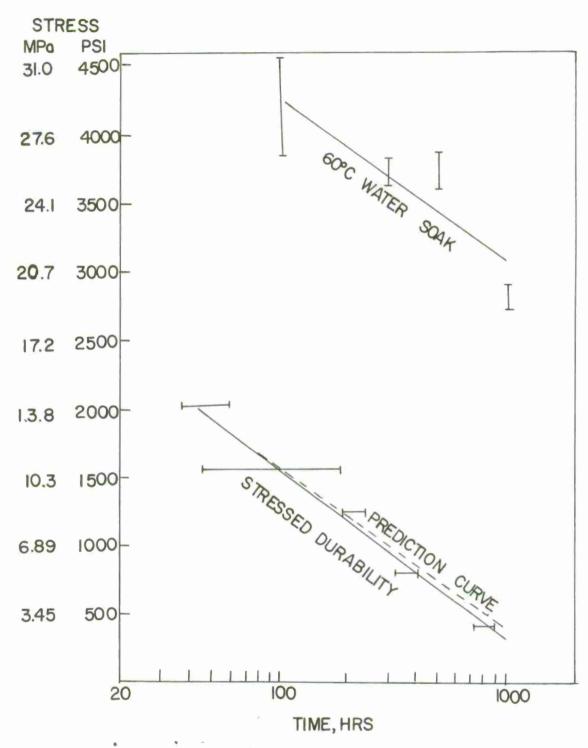


Figure 16. Comparison of 60°C.water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive J

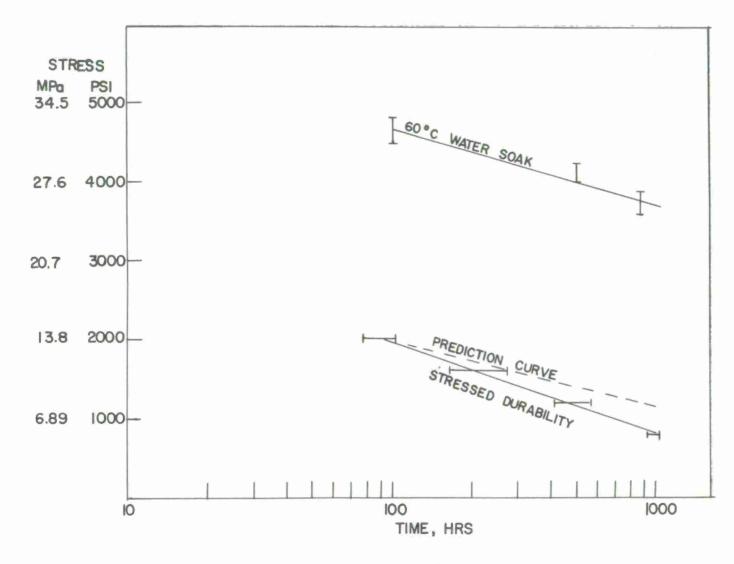


Figure 17. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for etched CP titanium alloy joints bonded with adhesive K

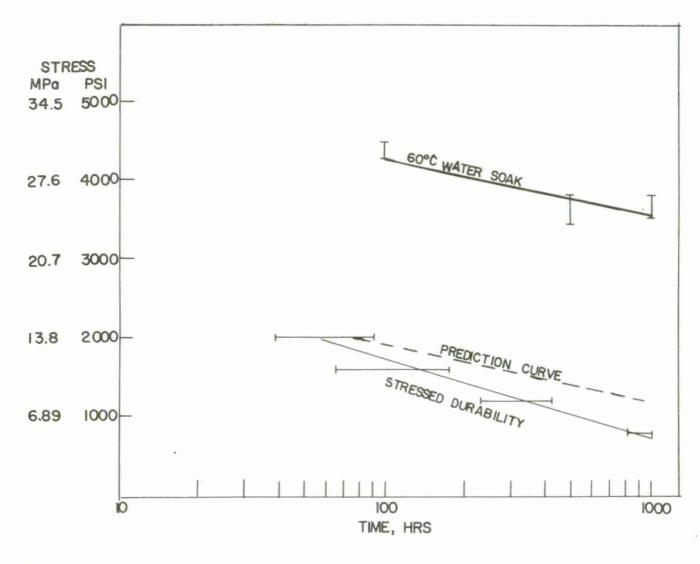


Figure 18. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for anodized 2024T3 aluminum alloy joints bonded with adhesive K

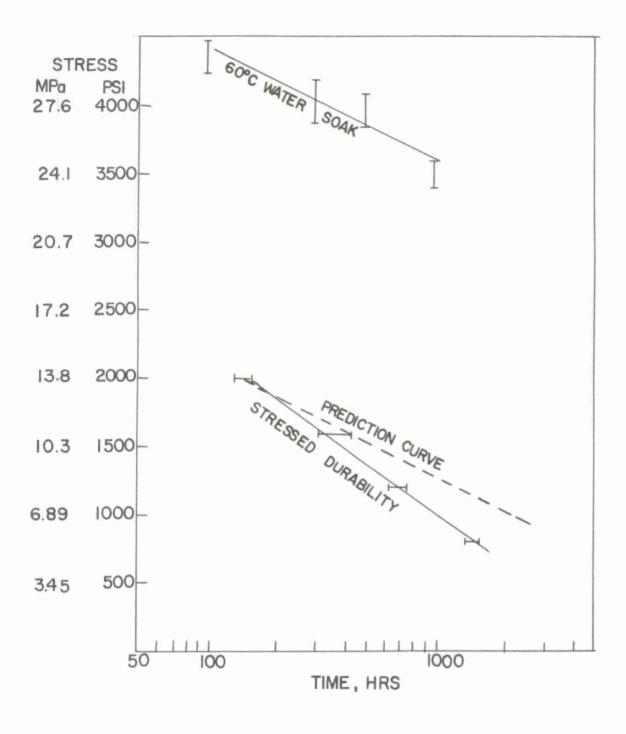


Figure 19. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive K

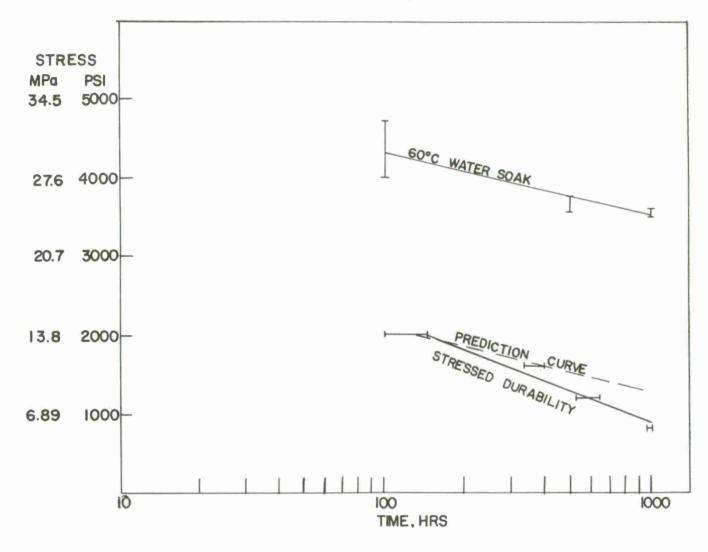


Figure 20. Comparison of 60°C water-soak residual-strength curves to stressed-durability curves for anodized 2024T3 aluminum alloy joints bonded with adhesive L

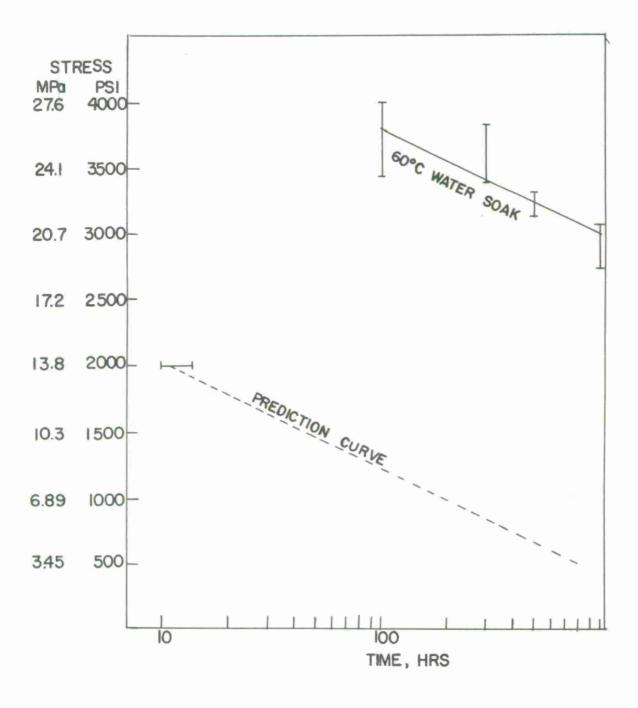


Figure 21. Comparison of 60°C water-soak residual-strength curves to predicted stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive E

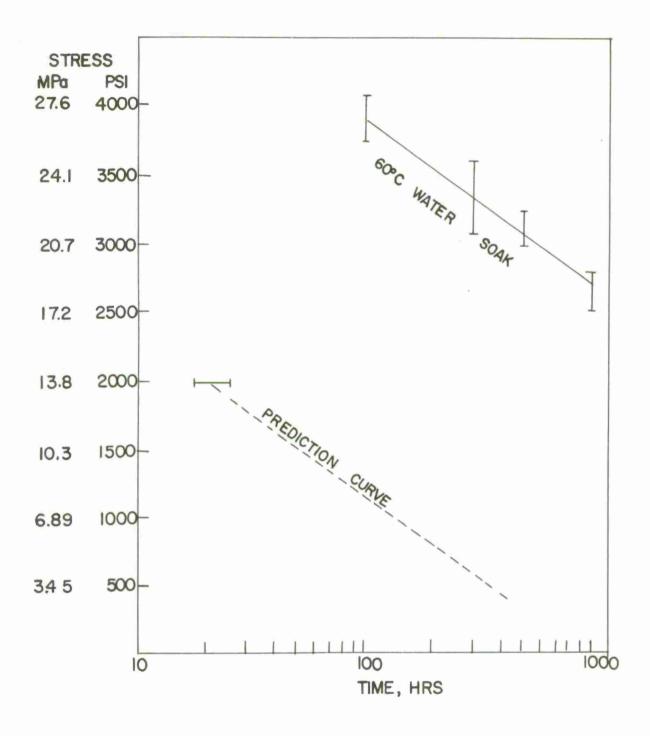


Figure 22. Comparison of 60°C water-soak residual-strength curves to predicted stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bended with adhesive F

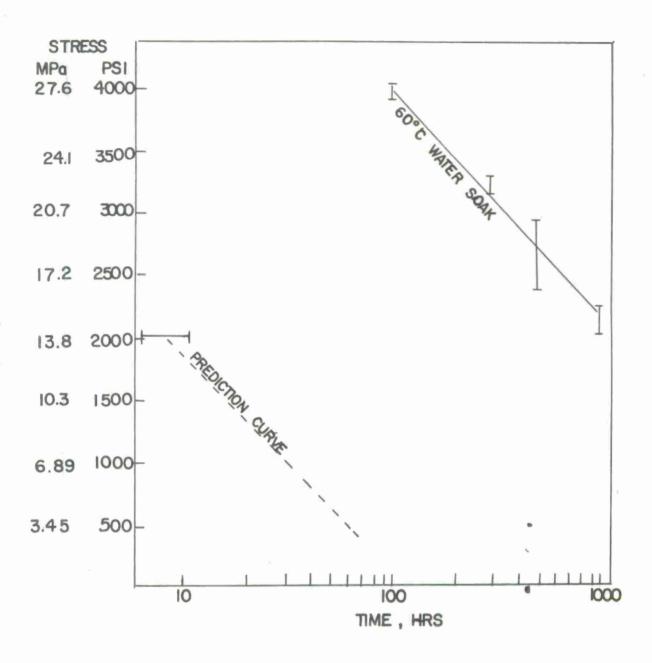


Figure 23. Comparison of 60°C water-soak residual-strength curves to predicted stressed-durability curves for FPL etched 2024T3 aluminum alloy joints bonded with adhesive H

Figure 24. Effect of -20°C storage of the adhesive on the durability of bonds tested by using the 1000 hour, 60°C water-soak residual-strength values

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